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U.S. PATENT APPLICATION

OF

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FOR

DRILLING FLUIDS CONTAINING AN ALKALI METAL FORMATE

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DRILLING FLUIDS CONTAINING AN ALKALI METAL FORMATE

5 BACKGROUND OF THE INVENTION

The present invention relates to the drilling industry and more particularly relates to drilling fluids used in drilling, such as the drilling of a well for the recovery of hydrocarbons or 10 other materials.

In drilling operations, such as the drilling that occurs in oil field operations, drilling fluids are designed/formulated to serve several functions. These functions include acting as a lubricant to the drill bit to reduce wear and friction during drilling and also to seal the formation surface by forming a filter cake. Currently, in the industry, both oil-based muds (OBMs) and water based muds (WBMs) are typically used. More commonly, synthetic based muds (SBMs) are also used in drilling operations. In the drilling fluid, agents for lubrication are present as well as weighting materials in order to achieve a density that typically produces a pressure greater than the surrounding pressure in the well bore. Furthermore, the drilling fluid will also contain a sealing or fluid loss agent, such as calcium carbonate for pore bridging especially polysaccharides and other 15 polymers, in order to form the filter cake on the formation surface of the well bore. In addition, when the drilling fluids are used during drilling, the drilling fluid will also contain drilling fines, such as shale and sandstone fines. During the drilling operations and afterwards, the filter cake seals the formation surface of the well bore so that the well bore can be completely formed without any leakage from the formation surface into the well bore and/or without any leakage of 20 the drilling fluids into the formation surface. While the filter cake is beneficial for these reasons, once the drilling is completed, and the recovery of hydrocarbons is the next step, the filter cake can act as a severe impediment to the recovery of hydrocarbons. For instance, the filter cake can prevent the recovery of hydrocarbons from the formation surfaces which have been blocked or sealed by the filter cake. Furthermore, when injectors are used to retain reservoir pressures, the 25 injection of sea water, for instance, can be significantly reduced due to the filter cake preventing the sea water from entering the formation and hence restricting the flow of water into the reservoir. Accordingly, the industry prefers to remove the filter cake from the well bore in order to 30

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optimize productivity. If the filter cake is not removed, the filter cake can block the pores that are part of the formation surface of the well bore which will interfere with the recovery of hydrocarbons. In many drilling operations, the drilling fluid can contain up to 5% by weight of a pore bridging material such as calcium carbonate. Calcium carbonate (CaCO₃) is typically a 5 blend of particle sizes with a particle size distribution designed to optimize the bridging of the pores found in the formation. The pore size distribution of the formation is determined from its permeability, preferably by direct porosity and permeability measurements of core plugs extracted from the reservoir.

Once drilling operations have been completed, the well is prepared for the completion 10 operations whereby the mud used for drilling is often displaced by a completion fluid. Completion fluids are typically water based clear fluids and are formulated to the same density as the mud used to drill the well in order to retain the hydraulic pressure on the well bore. There are numerous methods of completing a well, amongst which are open hole completions and gravel packed screened systems. The clear fluids are typically halide based brines such as calcium bromide, 15 calcium chloride, and zinc bromide; or organic based brines such as the formate based fluids.

In drilling an oil or gas well, the use of hydrocarbon-based drilling fluids are greatly preferred because of the inherent advantages of having an external phase fluid in contact with the formation. However, one severe disadvantage to a hydrocarbon-based drilling fluid is that weighting materials, such as barite, calcium carbonate, or hematite must be added to increase the 20 density of the fluid. These weighting-material solids are capable of inducing formation damage to producing formation.

Thus, there is a need to provide hydrocarbon-based drilling fluids that are preferably solids free or have low solids in the contents in the drilling fluid in order to avoid the above-mentioned disadvantages.

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SUMMARY OF THE PRESENT INVENTION

A feature of the present invention is to provide drilling fluids which are solids free or contain low amounts of solids in the drilling fluids.

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Another feature of the present invention is to provide drilling fluids which are a hydrocarbon-water emulsion which are suitable for use as drilling fluids.

A further feature of the present invention is to provide drilling fluids which can have a variety of different densities in order to be useful in a variety of drilling situations depending on 5 drilling depth and/or other variables.

Another feature of the present invention is to provide a more environmentally friendly drilling fluid that can be primarily aqueous based.

Additional features and advantages of the present invention will be set forth in part in the 10 description that follows, and in part will be apparent from the description, or may be learned by practice of the present invention. The objectives and other advantages of the present invention will be realized and attained by means of the elements and combinations particularly pointed out in the description and appended claims.

To achieve these and other advantages, and in accordance with the purposes of the present 15 invention, as embodied and broadly described herein, the present invention relates to a drilling fluid which contains at least one alkali metal formate and preferably at least one surfactant. Additional alkali metal formates, wetting agents, hydrocarbons, solid weighting materials, sealing or fluid loss agents, filtration control agents, and/or polymers to further control viscosity and/or other conventional additives such as organoclays and the like can also be optionally present for purposes of the present invention.

20 The present invention further relates to a method to drill a well comprising drilling of a well in the presence of the above-mentioned drilling fluid of the present invention.

The present invention further relates to a method to minimize or eliminate solids in a drilling fluid by substituting at least a portion of the solids weighting material with at least one alkali metal formate and preferably at least one surfactant.

25 It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are intended to provide a further explanation of the present invention, as claimed.

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DETAILED DESCRIPTION OF THE PRESENT INVENTION

The present invention relates to drilling fluids for use in drilling operations. For instance, the drilling fluid can be used in the drilling of a well for hydrocarbon recovery such as oil and/or gas. The drilling fluids of the present invention can also be used in other drilling operations where drilling fluids are used.

The drilling fluid of the present invention contains at least one alkali metal formate or monovalent carboxylic acid salt and preferably at least one surfactant or emulsifier. The drilling fluid of the present invention can contain an emulsion of an aqueous-based solution with a hydrocarbon-based fluid for purposes of forming the drilling fluid wherein the drilling fluid contains at least one alkali metal formate and preferably at least one surfactant along with at least one hydrocarbon-based fluid.

Preferably, the drilling fluid contains cesium formate as the alkali metal formate. More preferably, the drilling fluid contains two or more alkali metal formates, wherein preferably one of the alkali metal formates is cesium formate. A preferred combination of formates includes, but is not limited to, cesium formate with potassium formate. Other combinations of alkali metal formates can be used, such as sodium formate and potassium formate or sodium formate and cesium formate. Essentially, any combination of one or more monovalent carboxylic salts can be used for purposes of the drilling fluids of the present invention.

The alkali metal formates are commercially available. For instance, the cesium formate can be obtained from Cabot Corporation. The cesium formate can be made, for instance, by following the description as set forth in International Published Patent Application No. WO 96/31435, incorporated in its entirety by reference herein. The cesium formate that is present in the drilling fluid, preferably as a soluble salt, as stated above, can be present in any concentration and the cesium formate solution is a liquid at room temperature. Therefore, the concentration of the cesium formate in the drilling fluid can be from about 1% to about 100% by weight, and more preferably is present in an amount of from about 40% to about 95% by weight, and even more preferably is present in the drilling fluid at a range of from about 55% to about 85% by weight or is present in the drilling fluid at a range of from about 70% to about 85% by weight based on the

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weight of the drilling fluid. Besides the optional ingredients and preferably the surfactant and/or hydrocarbon fluid, the remainder of the drilling fluid can be water or other aqueous solutions. Conventional ingredients used in drilling fluids can also be used with the drilling fluid of the present invention.

5 Other alkali metal formates that can be used in the present invention are potassium formate and sodium formate which are commercially available. These alkali metal formates can also be prepared in a similar fashion as the cesium formate solution described above, and are also frequently obtained as by-products from ester hydrolysis.

Preferably, with respect to the drilling fluid of the present invention, at least 35 % by 10 volume of the fluid in the drilling fluid is an aqueous-based solution containing at least one alkali metal formate. More preferably, at least 50% by volume of the fluids present in the drilling fluid is an aqueous-based solution containing at least one alkali metal formate and even more preferably at least 75 % by volume of the fluids present in the drilling fluid of the present invention is an aqueous-based solution containing at least one alkali metal formate. Most preferably, at least 90 % 15 by volume of the fluids present in the drilling fluid of the present invention is an aqueous-based solution containing at least one alkali metal formate. In another embodiment, at least 95 % or more by volume of the fluids of the present invention contain an aqueous-based solution containing at least one alkali metal formate. In one embodiment, all of the fluids present are an aqueous-based solution containing at least one alkali metal formate wherein essentially no 20 hydrocarbon, or oil is present in the drilling fluid. Since the alkali formate is preferably dissolved in the aqueous solution, the drilling fluid can be solids free since the alkali formate preferably acts as a lubricant and a weighting material.

When a hydrocarbon fluid or synthetic mud fluid is present in the drilling fluids of the present invention, conventional hydrocarbon fluids or synthetic mud fluids can be used in the 25 drilling fluids of the present invention. Examples include, but are not limited to, diesel oil such as diesel oil number 2, crude oil, synthetic oils (such as paraffin oils, olefin oils, vegetable oils, and the like), as well as other conventional hydrocarbon fluids. Combinations of various hydrocarbon fluids or synthetic mud fluids can be used for purposes of the present invention. If a hydrocarbon or synthetic mud fluid is present in the drilling fluid of the present invention, various ratios of the

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hydrocarbon fluid to the aqueous-based solution described above can be used, such as ratios of 65 % by volume hydrocarbon fluid: 35 % aqueous based solution to 1 % by volume hydrocarbon fluid: 99 % by volume aqueous based solution.

When a hydrocarbon fluid is present with the aqueous-based solution containing at least 5 one alkali metal formate, at least one emulsifier or surfactant is preferably present in order to produce an emulsion of the ingredients. Essentially any emulsifier(s) or surfactant(s) capable of forming an emulsion between the hydrocarbon fluid and the aqueous based solution can be used for purposes of the present invention. Examples include, but are not limited to, a dimer trimer acid such as Witco DTA 350, imadazoline, tall oil (stearic acid), Integrity Synvert IV, Integrity Synvert 10 TWA, and the like. Any amount of surfactant or emulsifier can be used to form the emulsion such as from about 1 to about 30 pounds per barrel, wherein a barrel is about 42 gallons.

Other optional ingredients that can be present in the drilling fluids of the present invention include a filtration control agent or pore bridging materials such as Gilsonite and the like. These filtration control agents can be used in conventional amounts.

15 Other ingredients that can be present in the drilling fluids of the present invention include solid weighting materials such as barite, hematite, and/or calcium carbonate. These solid weighting materials can be used if desired. The amount of solid weighting material, which is optional, can be from about 0.5 pound per barrel to about 100 pounds per barrel.

Another optional ingredient in the drilling fluids of the present invention is a wetting agent 20 which can be helpful in emulsifying the alkali metal formate fluids with the hydrocarbon-based external fluids. An example of a suitable wetting agent is Integrity Synvert TWA. Conventional amounts can be used in combination with the emulsifiers described above in order to achieve desired emulsions of the formate fluids with the hydrocarbon-base external fluids.

Other ingredients that can optionally be present include, but are not limited to, other 25 drilling fluid products such as polymer(s) to add to viscosity, hydrophilic clays, fluid loss control additives, and the like. These other optional ingredients can be used in conventional amounts known to those skilled in the art.

The alkali metal formate that is present as part of the aqueous-based solution can be not fully saturated in the aqueous-based solution so as to permit any remaining water-soluble

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components to preferably solubilize in the solution along with the alkali metal formate. Thus, the alkali metal formate that is present in the aqueous-based solution can be present in an amount of less than 80 % by weight, based on the aqueous-based solution basis, and more preferably is from about 60 % to about 80 % by weight.

5 The important advantage of the present invention is the ability for the density of the drilling fluid to be adjusted to any desired density. This can especially be done with the introduction of a combination of alkali metal formates, such as potassium formate with cesium formate. As an example, an aqueous-based portion of the drilling fluid can contain cesium formate which can range from about 1.8 to about 2.4 s.g. This density range can be adjusted with the
10 introduction of potassium formate. For instance, when 0 to -100% by weight of potassium formate is included in the aqueous-based portion of the drilling fluid, the density of the overall aqueous-based portion of the drilling fluid can range from about 1.2 to about 2.4. Thus, the density of the drilling fluid can essentially be "dialed-in" to meet the density needed for the drilling fluid to be used in the drilling of the well bore at the appropriate depths. For lower density
15 ranges, sodium formate can be added to the potassium formate, hence, "dialing-in" lower density drilling fluids.

Thus, the drilling fluids of the present invention make it possible to achieve a variety of different densities and to minimize or completely eliminate the solid weighting material that is present in conventional drilling fluids.

20 The drilling fluids of the present invention can be introduced into the well bore by any conventional technique such as, but not limited to, being pumped into the drill pipe. Further, the drilling fluids can be recovered using conventional techniques.

25 The drilling fluids of the present invention can be prepared by mixing all of the components together. When an emulsion is prepared, typically, the components will be mixed together such as by shearing in order to ensure a dispersion that is preferably uniform with respect to the components.

For example, a typical parafinic hydrocarbon oil such as ESCAID 110, having a density of 8.03 s.g. (6.7 ppg) and a cesium formate solution having a density of 2.2 s.g. (18.36 ppg) when combined in a ratio of 1:1 and by addition of an emulsifier or a series of emulsifiers admixed by

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shearing, the fluids together can produce an emulsion or microemulsion that has a cesium formate invert phase and an oil external phase. The density of the combined mixture of this example is 1.51 s.g. (12.6 ppg).

For purposes of the present invention, when a hydrocarbon-based external fluid is used 5 with at least one formate fluid as described above, the formate fluids can be partially or totally emulsified into the hydrocarbon-based fluid. Or, in the alternative, when a majority of the drilling fluid is a formate fluid, the hydrocarbon-based fluid, if present, can be partially or totally emulsified into the formate fluid.

Optionally, the drilling fluids of the present invention can also contain at least one acid. 10 Preferably, the acid is an acid containing at least one carboxylic group and more preferably is formic acid or an acid derivative thereof. Other examples of acids that can be used include, but are not limited to, acetic acid, ascorbic acid, citric acid, tartaric acid, phthalic acid, glycolic acid, and combinations thereof. The acid can be present in various amounts such as from about 1 % or less to 25 weight % or more based on the weight of the drilling fluid. The presence of the acid has the 15 capability of adjusting the pH of the drilling fluid as well as providing other benefits to the drilling fluid. When an acid is present, for instance, the alkali metal formate fluid, such as cesium formate, can be present in any molar amount, but is preferably present in an amount of from about 3 M. Similarly, the acid, when present, can be present in any molar amount, but is preferably present in an amount of from about 2.2 M to about 15 M. The pH of the drilling fluid can be any pH.

20 The present invention will be further clarified by the following examples, which are intended to be purely exemplary of the present invention.

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EXAMPLES

Various drilling fluid formulations were prepared and tested to determine the ability of the drilling fluids to serve as suitable fluids for drilling.

In the Examples, cesium formate was used and as can be seen in the results summarized in 5 the various tables, a drilling fluid was made that had low or zero solids content and had the capability to "dial-in" various densities using a combination of components described herein.

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Cesium Formate, IA-35 (50/50 mixture)					
Mixing Procedure:					
1. Measure out IA-35, add Organoclay, mix 10 minutes				6. Hot Roll for 16 hrs at 150 F	
2. Add emulsifier(s), mix 5 minutes, add Gilsonite, mix 10 minutes					
3. Add CsF, mix 10 min. on HB, add CaCO ₃ ; mix 10 min. on HB				7. Remix, run rheology	
4. Run initial ES, viscosities at 120F				es, HPHT @ 250F	
5. Put 10 ml sample in a vial, SA for 24 hrs at 75F, measure volumes				8. Put vials in oven	
				SA 24hrs @ 150F	
Sample #	1	2	3	4	5
Integrity Synvert IV, lb/bbl	10	15	10	10	15
Dimer-trimer acid, lb/bbl	0	0	0	1	1
Integrity Synvert TWA, lb/bbl	0	0	0	6	6
Organoclay, lb/bbl	0	0	4	4	4
Gilsonite, lb/bbl	0	0	5	5	5
CaCO ₃ , lb/bbl	0	0	50	50	50
ES @ 120F	226	255	241	150	231
Hot-rolled 16 hrs, ES @ 120F	425	475	427	320	444
Initial Properties					
600 rpm @ 120F	39	42	162	181	155
300 rpm @ 120F	20	22	93	131	92
200 rpm @ 120F	14	15	66	110	67
100 rpm @ 120F	7	8	36	81	39
6 rpm @ 120F	0	1	3	33	6
3 rpm @ 120F	0	0	2	28	4
Plastic Viscosity, cP	19	20	69	50	63
Yield Point, lb/100ftsq	1	2	24	81	29
Gels, 10 sec/ 10 min.	0/0	0/2	3/7	29/32	7/7
HR 16 hrs @ 150F					
600 rpm @ 120F	51	55	169	195	188
300 rpm @ 120F	27	29	101	145	114
200 rpm @ 120F	14	14	75	113	86
100 rpm @ 120F	9	10	43	87	51
6 rpm @ 120F	1	1	4	37	8
3 rpm @ 120F	0	1	2	30	5
Plastic Viscosity, cP	24	26	68	50	74
Yield Point, lb/100ftsq	3	3	33	95	40
gels, 10 sec/ 10 min.	0/0	0/1	3/3	33/35	5/6
HPHT @ 250F, ml	28	22	15	14	6
Water in filtrate, ml	4	3	2	2	0
24 hrs @ 75F					
Free Oil %	5	10	4	3	2
Emulsion %	95	90	96	97	98
Free Brine %	0	0	0	0	0

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Cesium formate, 1A - 35 (30/50 minutes, 2.3 e.g. CsF)						2.3 e.g. CsF	559.24	331.48		
06/02/2000							50	18.52		
<i>Mixing Procedure:</i>										
1. Measure out 1A - 35							609.24	350	1.74099	14.44 2300
1A. Add Syntac, mix 5 minutes										
2. Add CsF, mix 10 min.										
3. Add Syntac 5, mix 10 minutes										
3A. Add calcium carbonate, mix 10 minutes										
4. Run initial ES, rheometrics at 120°F										
5. Put 10 ml sample in a vial, SA for 24 hrs at 75°F, measure volumes										
<i>NOTE: Sample 337 and 339 looked grainy with 15 lbs/bbl Symvert V. Concentration was increased to 28 lbs/bbl.</i>										
<i>Mud weight of 339 is 14.7 lbs/bbl (CsF in 337-340 is 2.37 e.g.)</i>										
<i>Sample #</i>		337	339	340	341	342	343	344		
1A - 35, grams		98.28	98.26	93.26	93.20	88.28	88.26	88.26	88.26	
Cesium Formate, grams		445	445	445	445	445	445	445	445	
Integrity Emulsifier, lbs/bbl		25	25	20	20	25	25	15	15	
Integrity Syntac, lbs/bbl		0.8	1	0.5	1	0.8	1	-	-	
Wilco DTA 350, lbs/bbl		-	-	-	-	-	-	0.5	1	
Banck Buregard 50, lbs/bbl		50	50	50	60	50	50	50	50	
Initial ES, rate	103	129	157	163	114	151	117	128		
Hei-rodded 16 hrs, ES							60			
<i>Initial Properties</i>		120°F	150°F	120°F	150°F	120°F	150°F	120°F	150°F	
800 rpm	162	116	186	118	164	115	183	128		
500 rpm	66	65	114	67	96	68	109	73		
200 rpm	67	47	63	49	70	48	80	53		
100 rpm	40	27	48	28	41	28	47	31		
6 rpm	6	7	8	6	7	6	8	8		
3 rpm	6	5	7	5	5	5	6	5		
<i>Plastic Viscosity, cP</i>		60	51	72	51	68	40	74	53	
Yield Pl, lb/100ft ²	24	14	42	16	28	17	35	20		
Gels, 10 sec/10 min.	6/7	6/6	6/7	5/6	6/8	5/5	6/7	5/3		
Setting in thermocup	no	no	no	no	no	no	no	no		
<i>HRR 30 hrs at 150°F</i>										
800 rpm at 150°F										
500 rpm										
200 rpm										
100 rpm										
6 rpm										
3 rpm										
<i>Plastic Viscosity, cP</i>										
Yield Pl, lb/100ft ²										
Gels, 10 sec/10 min.										
API R ₁ , ml total										
API R ₂ , ml oil										
API R ₃ , ml emulsion										
API R ₄ , ml brine										
HTHP at 180°F, ml										
HTHP at 250°F, ml										
250°F oil										
250°F emulsion										
250°F brine										
Comments										

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Cesium Formate, Escoid 110 (50/50 mixture)				
Mixing Procedure:	E-1	E-2	E-3	E-4
1. Measure out E-110, add organoclay, mix 10 minutes				
2. Add emulsifier(s), mix 5 minutes, add Gilsonite, mix 10 minutes				
3. Add CsF, mix 10 min. on HB, add CaCO ₃ , mix 10 min. on HB				
4. Run initial ES, viscoelasticities at 120F				
5. Put 10 ml sample in a vial, SA for 24 hrs at 75F, measure volumes				
6. Hot Roll for 18 hrs at 250F				
7. Remix, run rheology es, HPHT @ 250F				
8. Put vials in oven SA 24 hrs @ 150F				
Sample #	E-1	E-2	E-3	E-4
Integrity Synvert IV, lb/bbl	10	15	10	10
Dimer-trimer acid, lb/bbl	0	0	0	1
Integrity Synvert TWA, lb/bbl	0	0	0	6
Organoclay, lb/bbl	0	0	4	4
Gilsonite, lb/bbl	0	0	5	5
CaCO ₃ , lb/bbl	0	0	50	50
ES @ 120F	225	269	170	175
Hot-rolled 18 hrs, ES @ 120F	395	440	365	350
Initial Properties				
600 rpm @ 120F	28	30	85	92
300 rpm @ 120F	15	18	40	50
200 rpm @ 120F	10	11	31	41
100 rpm @ 120F	5	6	15	25
8 rpm @ 120F	0	1	3	6
3 rpm @ 120F	0	0	1	5
Plastic Viscosity, cP	13	14	45	42
Yield Point, lb/100ftsq	2	2	5	8
Gels, 10 sec/ 10 min.	0/0	0/1	1/4	5/8
HR 18 hrs @ 150F				
600 rpm @ 120F	31	33	96	110
300 rpm @ 120F	18	18	49	62
200 rpm @ 120F	10	12	35	53
100 rpm @ 120F	5	6	20	40
8 rpm @ 120F	0	2	4	10
3 rpm @ 120F	0	1	2	6
Plastic Viscosity, cP	15	15	47	48
Yield Point, lb/100ftsq	1	3	4	14
Gels, 10 sec/ 10 min.	0/0	1/1	3/4	7/8
HPHT @ 250F, ml	30	26	6	0
Water in filtrate, ml	4	3	0	0
24 hrs @ 75F				
Free Oil %	10	8	5	3
Emulsion %	90	92	95	97
Free Brin %	0	0	0	0

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Cesium Formate, IA-35 (75/25 mixture)					
Mixing Procedure:					
1. Measure out IA-35, add Organoclay, mix 10 minutes					
2. Add emulsifier(s), mix 5 minutes, add Gilsonite, mix 10 minutes					
3. Add CaF, mix 10 min. on HB, add CaCO3, mix 10 min. on HB					
4. Run initial ES, viscosities at 120F					
5. Put 10 ml sample in a vial, SA for 24 hrs at 75F, measure volumes					
6. Hot Roll for 16 hrs at 250F					
7. Remix, run rheology es, HPHT @ 250F					
8. Put vials in oven SA 24hrs @ 150F					
Sample #	1	2	3	4	5
Integrity Synvert IV, lb/bbl	10	15	10	10	15
Dimer-trimer acid, lb/bbl	0	0	0	1	1
Integrity Synvert TWA, lb/bbl	0	0	0	6	6
Organoclay, lb/bbl	0	0	4	4	4
Gilsonite, lb/bbl	0	0	5	5	5
CaCO3, lb/bbl	0	0	50	50	50
ES @ 120F	226	250	270	175	225
Hot-rolled 16 hrs, ES @ 120F	185	200			
Initial Properties					
800 rpm @ 120F	251	265	t	t	t
300 rpm @ 120F	169	172	0	0	0
200 rpm @ 120F	135	133	0	0	0
100 rpm @ 120F	92	84			
6 rpm @ 120F	26	13	t	t	t
3 rpm @ 120F	20	8	h	h	h
Plastic Viscosity, cP	82	93	c	c	c
Yield Point, lb/100ftsq	87	79	k	k	k
Gels, 10 sec/ 10 min.	24/28	8/12			
HR 16 hrs @ 150F					
800 rpm @ 120F	278	259			
300 rpm @ 120F	175	166			
200 rpm @ 120F	140	101			
100 rpm @ 120F	99	22			
6 rpm @ 120F	29	14			
3 rpm @ 120F	22	8			
Plastic Viscosity, cP	101	93			
Yield Point, lb/100ftsq	74	73			
gels, 10 sec/ 10 min.	22/23	8/12			
HPHT @ 250F, ml	23	16			
Water In filtrate, ml	8	4			
24 hrs @ 75F					
Free Oil %	1	0	0		
Emulsion %	99	100	100		
Free Brine %	0	0	0		

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24 hrs @ 150F							
Free Oil %		2	2	0			
Emulsion %		98	98	100			
Free Brine %		0	0	0			

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Observational Notes:

- Emulsion is clear at temperatures above 130 F
- Amount of shear imparted to system is directly related to emulsion stability
- Heating the emulsion to 150 F acts to stabilize emulsion (similar to shearing)
- Oil / Water ratios as low as 25/75 are achievable with this system

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Cesium Formate, IA-35 (50/50 mixture)					
Mixing Procedure:					
1. Measure out IA-35, add Organoclay, mix 10 minutes					6. Hot Roll for 16 hrs at 150 F
2. Add emulsifier(s), mix 5 minutes, add Gilsonite, mix 10 minutes					
3. Add CsF, mix 10 min. on HB, add CaCO ₃ , mix 10 min. on HB					7. Remix, run rheology es, HPHT @ 250F
4. Run initial ES, viscosities at 120F					
5. Put 10 ml sample in a vial, SA for 24 hrs at 75F, measure volumes					8. Put vials in oven SA 24hrs @ 150F
Sample #	1	2	3	4	5
Integrity Synvert IV, lb/bbl	10	15	10	10	15
Dimer-trimer acid, lb/bbl	0	0	0	1	1
Integrity Synvert TWA, lb/bbl	0	0	0	6	6
Organoclay, lb/bbl	0	0	4	4	4
Gilsonite, lb/bbl	0	0	5	5	5
CaCO ₃ , lb/bbl	0	0	50	50	50
ES @ 120F	228	255	241	150	231
Hot-rolled 16 hrs, ES @ 120F	425	475	427	320	444
Initial Properties					
600 rpm @ 120F	39	42	162	181	155
300 rpm @ 120F	20	22	93	131	92
200 rpm @ 120F	14	15	66	110	67
100 rpm @ 120F	7	8	36	81	39
6 rpm @ 120F	0	1	3	33	6
3 rpm @ 120F	0	0	2	28	4
Plastic Viscosity, cP	19	20	69	50	63
Yield Point, lb/100ftsq	1	2	24	81	29
Gels, 10 sec/ 10 min.	0/0	0/2	3/7	29/32	7/7
HR 16 hrs @ 150F					
600 rpm @ 120F	51	55	169	195	183
300 rpm @ 120F	27	29	101	145	114
200 rpm @ 120F	14	14	75	113	85
100 rpm @ 120F	9	10	43	87	51
6 rpm @ 120F	1	1	4	37	8
3 rpm @ 120F	0	1	2	30	5
Plastic Viscosity, cP	24	26	88	50	74
Yield Point, lb/100ftsq	3	3	33	95	40
gels, 10 sec/ 10 min.	0/0	0/1	3/3	33/35	5/6
HPHT @ 250F, ml	28	22	15	14	6
Water in filtrate, ml	4	3	2	2	0
24 hrs @ 75F					
Free Oil %	5	10	4	3	2
Emulsion %	95	90	96	97	98
Free Brine %	0	0	0	0	0

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24 hrs @ 150F								
Free Oil %		18	15	10	6			
Emulsion %		82	85	90	94			
Free Brine %		0	0	0	0			

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Cesium formate, Escald 110 (50/50 mixture)											
Mixing Procedure:											
1. Measure out Escald 110, add Gehane II, mix 10 minutes							176 ml Escald 110 (0.603 sg) - (140.5 grams)				
2. Add emulsifiers (Below in bold), mix 5 minutes, add Barablock, mix 10 minutes							175 ml Cesium Formate (2.2 sg) - (385 grams)				
3. Add Cet, mix 10 min. on HB, add Lo-Wate, mix 10 min. on HB											
4. Run initial ES, viscosities at 120°F											
5. Put 10 ml sample in a vial, SA for 24 hrs at 75°F, measure volumes											
Sample #	225	226	227	228	229	230	231	232	233	234	235
Integrity Synvert II (Lots 991512), lb/bbl	7	7	7	7	7	7	7	7	7	7	7
Witco DTA 350 (dimer-trimer), lb/bbl	1	1	1	1	1	1	1	1	1	1	1
Integrity Synvert TWA (1/24/00, 0116), lb/bbl	2	4	6	2	4	6	2	4	6	2	4
Baroid Gelstone R, lb/bbl	-	-	-	4	4	4	-	-	4	4	4
Baroid Barsblock 400, lb/bbl	-	-	-	-	-	5	5	5	5	5	5
M-I Lo-Wate, lb/bbl	50	50	50	50	50	50	50	50	50	50	50
Initial ES, volts	107	194	247	127	123	128	139	263	249	105	133
Hot-rolled 16 hrs, ES	426	450	290	150	185	170	402	314	307	144	196
Initial Properties											
600 rpm at 75°F	80	67	30	99	74	87	81	90	85	86	81
300 rpm	58	48	60	75	52	90	63	68	85	82	80
200 rpm	49	40	61	84	43	48	62	59	55	50	47
100 rpm	37	31	39	49	31	35	40	46	43	37	34
6 rpm	13	10	13	18	12	13	17	14	14	12	14
3 rpm	10	6	4	15	10	11	12	5	6	10	12
Plastic Viscosity, cP											
Yield Pt, lb/100ft ²	22	19	20	24	22	27	18	21	20	24	24
Gels, 10 sec/10 min.	38	29	40	51	30	33	45	48	46	38	33
	11/7	5/7	7/3	16/19	10/10	10/11	17/15	10/5	11/6	10/11	12/13
HTHP 16 hrs at 150°F											
600 rpm at 120°F	71	89	95	84	75	70	111	129	122	63	80
300 rpm	53	69	74	68	52	48	84	92	86	43	62
200 rpm	44	69	63	58	43	39	71	78	63	35	50
100 rpm	34	45	48	42	31	28	63	61	65	25	37
6 rpm	10	12	12	17	12	10	17	18	21	8	13
3 rpm	4	5	4	13	10	8	7	7	7	11	14
Plastic Viscosity, cP											
Yield Pt, lb/100ft ²	18	20	21	26	23	22	27	26	26	20	28
Gels, 10 sec/10 min.	35	49	53	42	29	28	57	66	70	23	34
	7/4	6/4	10/4	12/13	10/11	9/9	13/5	12/6	13/5	7/8	10/11
API Shear, ml	0.6	1.1	1.1	1.6	0.9	0.8	1.2	1.2	0.3	0.2	0.0
API Filtrate, ml	0	0	0	0	0	0	0	0	0	0	0
API Filtrate, brine	0	0	0	0	0	0	0	0	0	0	0
HTHP at 150°F, ml	8.6	3.0	5.0	3.5	1.6	0.8	2.6	2.0	1.4	2.0	trace
Water in filtrate?	6.0	2.0	2.6	1.5	0.4	0.0	0.0	0.0	0.0	0.0	0.0
HTHP at 250°F, ml	24.0	19.0	12.0	8.2	4.2	2.5	8.0	8.0	6.0	7.0	5.0
Water in filtrate?	12.0	9.0	3.1	2.6	1.7	0.6	0.0	0.0	0.0	0.0	0.0
24 hrs at 75°F											
Total fluid height	34	35	34	35	36	33	35	35	35	34	34
free oil height	6	3	2	1	2	3	2	3	2	1	1
emulsion height	28	32	32	34	35	31	32	33	32	33	33
free brine height	0	0	0	0	0	0	0	0	0	0	0
free oil, %	18%	9%	8%	3%	3%	6%	9%	8%	9%	8%	3%
emulsion, %	82%	91%	94%	97%	97%	84%	91%	95%	81%	94%	97%
free brine, %	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%
24 hrs at 150°F											
Total fluid height	34	36	35	35	37	32	35	38	35	34	35
free oil height	8	6	8	3	3	4	7	6	6	6	4
emulsion height	28	28	29	32	34	28	28	30	28	31	32
free brine height	0	0	0	0	0	0	0	0	0	0	0
free oil, %	24%	22%	17%	9%	8%	13%	20%	17%	18%	11%	9%
emulsion, %	76%	78%	67%	91%	92%	88%	80%	83%	82%	89%	91%
free brine, %	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%
Difference (72 hrs/24 hrs at 150°F)											
free oil, %	6%	14%	11%	6%	5%	6%	11%	11%	9%	12%	8%
emulsion, %	-6%	-14%	-11%	-6%	-5%	-8%	-11%	-11%	-9%	-12%	-8%
free brine, %	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%

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Sample #	Mixing Order:				
	1	2	3	4	5
IA-35, %	90	75	50	25	10
Synvert IV, lb/bbl	20	20	20	20	20
2.2 s.g. Cs Formate, %	10	25	50	75	90
Initial Rheologies					
Mud weight, lb/gal	7.81	9.4	11.94	14.47	17.18
600 rpm at 120°F	7	13	42	>300	too thick
300 rpm	4	7	21	274	to measure
200 rpm	3	5	14	218	
100 rpm	2	3	7	149	
6 rpm	0.2	0.5	1	38	
3 rpm	0.2	0.5	0.5	28	
PV	3	6	21		
YP	1	1	0		
Gels	5/5	1/1	1/1	28/32	
ES	373	310	362	328	133
Vials 16 hrs at 75°F					
Total Height	32	32	32	31	
Oil	18	1	0	0	
Emulsion	14	31	32	31	
Formate					
Samples H.R. for 16 hours at 150°F					
600 rpm at 120°F	6	13	45	>300	too thick
300 rpm	3	7	23	273	to measure
200 rpm	2	4	16	222	
100 rpm	1	2	8	158	
6 rpm	0	0	2	49	
3 rpm	0	0	2	40	
PV	3	6	22		
YP	0	1	1		
Gels	1/1	0/0	2/2	38/42	
ES	1069	211	232	271	115
Vials 7 hrs at 150°F					
Total Height	32	32	32	32	
Oil	22	3	1	0	
Emulsion	10	29	31	32	
Formate					
Vials 64 hrs at 200°F					
Total Height	105	103	100	100	105
Oil	89	66	40	0	0
Emulsion	8	12	60	100	105
Formate	8	25	0	0	0
Vials 64 hrs at 250°F					
Total Height	103	100	100	100	110
Oil	95	70	30	32	0
Emulsion	8	7	65	60	110
Formate	0	23	5	8	0
Vials 64 hrs at 300°F					
Total Height	102	105	102	102	104
Oil	92	70	42	32*	19*
Emulsion	10	10	10		
Formate	0	25	50	70	85

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Integrity Synvert IV Sample 1 gallon can	Test Procedure		
	1. Mix sample		
	2. Place aliquots of sample in large test tubes		
	3. Static-age samples for 24 hours at 200, 250, and 300°F, 300 psi N2		
	4. Static-age samples for 72 hours at 200, 250, and 300°F, 300 psi N2		
5. Measure total height, oil, emulsion, and brine			
24 hour tests			
	200°F	250°F	300°F
Total height, mm	100	99	99
Free Oil, mm	9	16	30
Emulsion, mm	84	73	46
Free Brine, mm	7	10	23
% Oil	9.0%	16.2%	30.3%
% Emulsion	84.0%	73.7%	46.5%
% Brine	7.0%	10.1%	23.2%
72 hour tests			
	200°F	250°F	300°F
Total height, mm	96	101	97
Free Oil, mm	5	10	30
Emulsion, mm	81	71	22
Free Brine, mm	10	20	45
% Oil	5.2%	9.9%	30.9%
% Emulsion	84.4%	70.3%	22.7%
% Brine	10.4%	19.8%	46.4%
Note: the 24 hr 300°F, and the 72 hour 200 and 250°F fluids appeared to have two phases in the emulsion portion. One phase is more distinctly oil and the second phase is more distinctly emulsion.			

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Sample of 50/50 mix from Integrity (one gallon can)

600 rpm at 75°F	90
300 rpm	49
200 rpm	34
100 rpm	18
6 rpm	2
3 rpm	1

PV	41
YP	8
Gels	2/2
ES	457

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Integrity Turpene-Formate Sample
~~12/17/03 08:12~~

Vials 3 and 7 days at 200°F	<u>3 days</u>	<u>7 days</u>
Total Height	50	52
Oil	20	15
Emulsion	0	0
Formate	30	37

Vials 3 and 7 days at 250°F	<u>3 days</u>	<u>7 days</u>
Total Height	54	51
Oil	17	15
Emulsion	0	0
Formate	37	36

Vials 3 and 7 days at 300°F	<u>3 days</u>	<u>7 days</u>
Total Height	51	50
Oil	15	15
Emulsion	0	0
Formate	36	35

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	SA 250			SA 250			353
	Initial	349.5	349.75	350	350.5	350.75	
<u>Sample #</u>	<u>349</u>	<u>349.5</u>	<u>349.75</u>	<u>350</u>	<u>350.5</u>	<u>350.75</u>	<u>353</u>
IA - 35, grams	94.26	94.26	94.26	94.26	94.26	94.26	89.26
Cesium Formate, grams	445	445	445	445	445	445	445
Integrity Emulsifier, lb/bbl	20	20	20	20	20	20	25
Integrity Synvis, lb/bbl	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Witco DTA 350, lb/bbl	-	-	-	0.5	0.5	0.5	-
Baroid Barablock, lb/bbl	-	-	-	-	-	-	-
M-I Lo Wate, lb/bbl	50	50	50	50	50	50	50
Initial ES, volts	227			256			301
Hot-rolled 16 hrs, ES	109			209			339
Static-aged ES, volts		113			154		
<u>Initial Properties</u>	<u>120°F</u>	<u>120°F</u>	<u>120°F</u>	<u>120°F</u>	<u>120°F</u>	<u>120°F</u>	<u>120°F</u>
600 rpm	188	147	-	179	148	-	203
300 rpm	113	90	-	108	88	-	122
200 rpm	83	68	-	80	65	-	90
100 rpm	49	41	-	48	39	-	53
6 rpm	8	6	-	6	6	-	7
3 rpm	5	4	-	4	4	-	5
Plastic Viscosity, cP	75	57	-	71	60	-	81
Yield Pt, lb/100ft ²	38	33	-	37	28	-	41
Gels, 10 sec/10 min.	5/6	5/6	-	4/5	4/6	-	6/6
Settling in thermocup	no	no	-	no	no	-	no
Initial ES, volts	253			266			295
Hot-rolled 16 hrs, ES	205	145	331	222	175	330	242
HR 16 hrs at 150°F	<u>150°F</u>	<u>SA 250</u>	<u>SA 250</u>	<u>150°F</u>	<u>SA 250</u>	<u>SA 250</u>	<u>150°F</u>
		Initial	Silv.		Initial	Silv.	
600 rpm	137	98	121	137	104	121	158
300 rpm	81	58	70	82	60	70	95
200 rpm	60	42	51	61	44	51	70
100 rpm	36	25	30	37	26	30	42
6 rpm	7	4	5	7	5	5	8
3 rpm	5	3	3	5	3	3	6

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Plastic Viscosity, cP	56	40	51	55	44	51	63
Yield Pt, lb/100ft^2	25	18	19	27	16	19	32
Gels, 10 sec/10 min.	6/6	3/3	3/4	5/5	3/4	3/4	6/6
API filt, ml total	0.7	-	-	0.4	-	-	0.3
API filt, ml oil	0	-	-	0	-	-	0
API filt, ml emulsion	0.7	-	-	0.4	-	-	0.3
API filt, ml brine	0.0	-	-	0.0	-	-	0.0
HTHP at 250°F, ml	3.2	-	1.6	8.8	-	2.0	4.8
250°F oil	2.4	-	-	6	-	-	3.6
250°F emulsion	0.8	-	1.6	2.8	-	2.0	1.2
250°F brine	-	-	-	-	-	-	-
Free Oil, %				22%			25%

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SA 250 Initial	SA 250 Silv.	354	SA 250 Initial	SA 250 Silv.
353.5	353.75	354	354.5	354.75
89.26	89.26	89.26	89.26	89.26
445	445	445	445	445
25	25	25	25	25
0.5	0.5	0.5	0.5	0.5
-	-	0.5	0.5	0.5
-	-	-	-	-
50	50	50	50	50

384

306

150		240	
<u>120°F</u>	<u>120°F</u>	<u>120°F</u>	<u>120°F</u>
159	-	224	205
97	-	138	125
73	-	103	93
44	-	65	56
7	-	10	9
5	-	7	7

62	-	86	80	-
35	-	52	45	-
6/7	-	7/8		-
no	-	no	no	-

387

161 369 314 218 336

SA 250 Initial	SA 250 Silv.	150°F	SA 250 Initial	SA 250 Silv.
<u>150°F</u>	<u>150°F</u>	<u>150°F</u>	<u>150°F</u>	<u>150°F</u>
114	136	167	133	157
68	80	102	78	94
50	59	76	57	69
30	35	47	34	41
5	7	10	6	6
4	5	8	4	4

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46	56	65	55	63
22	24	37	23	31
4/5	5/6	8/8	5/5	5/5
-	-	1.8	-	-
-	-	0.1	-	-
-	-	1.7	-	-
-	-	0.0	-	-
-	2.4	3.6	-	4.8
-	-	0.4	-	0.3
-	1.0	3.2	-	2.5
-	1.4	-	-	2.0
	25%		25%	

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Sample #	351	355	359			
IA - 35, grams	94.26	89.26	84.26			
Cesium Formate, grams	445 (2.3)	445 (2.3)	445 (2.3)			
Integrity Emulsifier, lb/bbl	20	25	30			
Integrity Synvis, lb/bbl	0.5	0.5	0.5			
Witco DTA 350, lb/bbl	0.5	0.5	0.5			
Baroid Barablock, lb/bbl	10	10	10			
M-I Lo Wate, lb/bbl	50	50	50			
Initial ES, volts	301	300	357	365	440	492
Hot-rolled 16 hrs, ES	308	348	328	325	368	389
Initial Properties	120°F	150°F	120°F	150°F	120°F	150°F
600 rpm	195	193	241	227	300+	300+
300 rpm	117	120	147	143	253	233
200 rpm	87	91	111	110	196	182
100 rpm	53	56	70	70	127	119
6 rpm	7	10	12	15	28	29
3 rpm	5	7	8	11	20	22
Plastic Viscosity, cP	78	73	94	84	-	-
Yield Pt, lb/100ft^2	39	47	53	59	-	-
Gels, 10 sec/10 min.	5/6	8/9	8/10	11/14	19/22	22/26
Settling in thermocup	no	no	1	2	9	10
HR 16 hrs at 150°F	120°F	150°F	120°F	150°F	120°F	150°F
600 rpm	300+	226	300+	274	300+	300+
300 rpm	201	140	246	171	300+	242
200 rpm	154	106	188	129	282	186
100 rpm	98	67	119	81	181	121
6 rpm	20	15	25	19	42	32
3 rpm	15	11	19	15	32	26
Plastic Viscosity, cP	-	86	-	103	-	-
Yield Pt, lb/100ft^2	-	54	-	68	-	-
Gels, 10 sec/10 min.	14/19	11/12	18/22	14/15	32/33	25/27
API filt, ml total	0		0.0		0.0	
API filt, ml oil	-		0.0		0.0	
API filt, ml emulsion	0		0.0		0.0	
API filt, ml brine	-		0.0		0.0	
HTHP at 150°F, ml						
HTHP at 250°F, ml	trace		1.2		trace	
250°F oil			0.0		0.0	
250°F emulsion			1.2		trace	
250°F brine			0.0		0.0	
Comments						

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Sample #	355	356	357	358	359	360	
IA - 35, grams	89.26	89.26	84.26	84.26	84.26	84.26	
Cesium Formate, grams	445 (2.3)	445 (2.3)	445 (2.3)	445 (2.3)	445 (2.3)	445 (2.3)	
Integrity Emulsifier, lb/bbl	25	25	30	30	30	30	
Integrity Synvis, lb/bbl	0.5	0.5	0.5	0.5	0.5	0.5	
Wilco DTA 350, lb/bbl	0.5	1.0		0.5	0.5	1.0	
Baroid Barablock, lb/bbl	10				10		
M-3 Lo Wate, lb/bbl	50	50	50	50	50	50	
Initial ES, volts	357	365	329	311	335	316	344
Hot-rolled 16 hrs, ES	328	325	307	307	298	301	301
Initial Properties	120°F	150°F	120°F	150°F	120°F	150°F	120°F
600 rpm	241	227	196	154	235	182	245
300 rpm	147	143	121	95	147	110	150
200 rpm	111	110	92	72	108	82	113
100 rpm	70	70	59	46	67	51	70
6 rpm	12	15	11	10	13	11	13
3 rpm	8	11	8	7	10	8	9
Plastic Viscosity, cP	94	84	75	59	88	72	95
Yield Pt, lb/100ft^2	53	59	46	36	59	38	55
Gels, 10 sec/10 min.	8/10	11/14	8/9	7/8	9/10	9/9	9/10
Settling in thermocup	1	2	3	4	5	6	7
HR 16 hrs at 150°F	120°F	150°F	120°F	150°F	120°F	150°F	120°F
600 rpm	300+	274	201	153	229	174	245
300 rpm	246	171	124	94	140	106	151
200 rpm	188	129	95	71	105	79	114
100 rpm	119	81	60	49	65	49	70
6 rpm	25	19	12	10	13	12	14
3 rpm	19	15	9	8	10	9	10
Plastic Viscosity, cP	-	103	77	59	89	68	94
Yield Pt, lb/100ft^2	-	68	47	35	51	38	57
Gels, 10 sec/10 min.	18/22	14/15	9/10	8/8	10/10	9/10	10/11
API filt, ml total	0.0		1.8		0.0	1.0	0.0
API filt, ml oil	0.0		0.1		0.0	trace	0.0
API filt, ml emulsion	0.0		1.7		0.0	1.0	0.0
API filt, ml brine	0.0		0.0		0.0	0.0	0.0
HTHP at 150°F, ml							
HTHP at 250°F, ml	1.2		7.4		4.8	4.4	trace
250°F oil	0.0		0.0		0.0	0.0	0.0
250°F emulsion	1.2		7.4		4.8	4.4	trace
250°F brine	0.0		0.0		0.0	0.0	2.0
Comments							

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Cesium formate, IA - 35 (50/50 mixture, 2.3 s.g. CsF)										2.3 sg csf	559.24	331.48
Mixing Procedure:										50	18.52	
1. Measure out IA - 35										609.24	350	1.74069 14.4999
1A. Add Synvis, mix 5 minutes												
2. Add CsF, mix 10 min.												
3. Add Synvert 5, mix 10 minutes												
3A. Add calcium carbonate, mix 10 minutes												
4. Run initial ES, viscosities at 120°F												
5. Put 10 ml sample in a vial. SA for 24 hrs at 75°F, measure volumes												
41.6/58.4 oil/csF ratio										347	348	
Sample #	337	338	339	340	347	348						
IA - 35, grams	98.26	98.26	93.26	93.26	88.26	88.26						
Cesium Formate, grams	445 (2.366)	445 (2.366)	445 (2.366)	445 (2.366)	445 (2.3)	445 (2.3)						
Integrity Emulsifier, lb/bbl	25	25	20	20	25	25						
Integrity Synvis, lb/bbl	0.5	1	0.5	1	-	-						
Wilco DTA 350, lb/bbl	-	-	-	-	0.5	1						
Baroid Baracarb 50, lb/bbl	50	50	50	50	50	50						
Initial ES, volts	103	129	167	166	117	128	212	192	205	243		
Hot-rolled 16 hrs. ES	114	102	148	140	139	138	122	118	145	252	247	
Initial Properties	120°F	150°F	120°F	150°F	120°F	150°F	120°F	150°F	120°F	150°F	120°F	150°F
600 rpm	162	116	186	118	164	115	183	126	177	132	182	132
300 rpm	93	65	114	67	96	66	109	73	106	78	111	80
200 rpm	67	47	83	49	70	48	80	53	77	57	83	61
100 rpm	40	27	49	29	41	28	47	31	46	347	52	38
6 rpm	8	7	9	6	7	6	8	6	8	7	10	8
3 rpm	6	5	7	5	5	5	6	5	6	5	7	6
Plastic Viscosity, cP	69	51	72	51	68	49	74	53	71	54	71	52
Yield Pt, lb/100ft ²	24	14	42	16	28	17	35	20	35	24	40	28
Gels, 10 sec/10 min.	6/7	6/6	6/7	5/6	5/5	6/7	5/5	6/6	6/6	6/6	8/8	6/7
Settling in thermocup	no	no	no	no	no	no	no	no	no	no	no	no
HR 16 hrs at 150°F	120°F	150°F	120°F	150°F	120°F	150°F	120°F	150°F	120°F	150°F	120°F	150°F
600 rpm	161	113	187	123	162	114	184	129	179	140	184	144
300 rpm	93	64	116	70	97	66	111	75	109	84	113	88
200 rpm	68	46	86	52	72	48	82	55	80	62	85	66
100 rpm	39	27	52	30	473	28	49	32	48	37	52	41
6 rpm	8	6	10	6	7	6	8	6	8	7	10	9
3 rpm	6	5	8	5	5	4	6	5	6	5	8	7
Plastic Viscosity, cP	68	49	71	53	65	48	73	56	70	56	71	56
Yield Pt, lb/100ft ²	25	15	45	17	32	18	38	28	39	32	42	32
Gels, 10 sec/10 min.	6/6	5/6	7/9	5/6	6/6	5/5	6/7	5/6	6/6	5/5	8/8	7/9
API filt. ml total	1.0		0.2		0.4		0.3		3.5		6.2	
API filt. ml oil												
API filt. ml emulsion	1.0		0.2		0.4		0.3		3.5		6.2	
API filt. ml brine												
HTHP at 150°F, ml					0.2		0.2		19.4	(0.8 brine)		
HTHP at 250°F, ml					11.4		4.4					
250°F oil					0.0		0.0					
250°F emulsion					6.8		3.2					
250°F brine					4.6		1.2					
Comments												
NOTE:	1. Sample 337 and 338 looked grainy with 15 lb/bbl Synvert V. Concentration was increased to 25 lb/bbl. After hot-rolling at 150°F, #337 and #338 still appear grainy. 2. Mud weight of 339 is 14.7 lb/gal (CsF in 337-340 is 2.366 s.g.) 3. Mud weight of 347 is 14.6 lb/gal (CsF in 347-348 is 2.30 s.g.)											

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Cesium formate, Escaid 110 (50/50 mixture)												
175 ml Escaid 110 (0.803 sg) - (140.5 grams) 175 ml Cesium Formate (2.2 sg) - (365 grams)												
Mixing Procedure:												
1. Measure out Escaid 110												
2. Add emulsifiers, mix 5 minutes												
3. Add CsF, mix 10 min, on HB, mix on Silverson to 135°F												
3A. Add Lo-Wate, mix 10 min, on HB												
4. Run initial ES, viscosities at 120°F												
5. Put 10 ml sample in a vial, SA for 24 hrs at 75°F, measure volumes												
6. Hot-roll for 16 hours at 150°F												
7. Remix, run rheology, ES, API, HTHP at 150°F												
8. Put vials in oven and static-age at 150°F for 24 hrs, measure volumes												
9. If 150°F HTHP is good, run at 250°F												
Sample #	273	274	275	276	277	278	279	280	281	282	283	284
Integrity Synver IV (Lot# 000329), lb/bbl	5	10	10	10	15	15	5	5	5	5	2.5	2.5
Witco AX-180-2, lb/bbl	5	5	5	5	5	5	7.5	10	10	10	10	10
Witco DTA 350, lb/bbl dimer trimer			2	3		2					2	
Baroid Baracarb 50, lb/bbl	50	50	50	50	50	50	50	50	50	50	50	50
Initial ES, volts @ 120°F	363	380	425	572	454	417	448	390	445	582	360	458
Hot-rolled 16 hrs, ES	399	410	378	489	403	329	398	431	345	288	359	229
Jar - S = Setting, 3 = 3 Phases, N = No	S	S	S	S	S	S	S	S	N	N	S	S
Initial Properties												
600 rpm at 120°F	24	25	29	42	27	29	26	24	30	47	24	32
300 rpm	13	14	17	30	15	15	14	13	18	33	13	19
200 rpm	9	9	12	25	10	11	9	9	13	28	9	15
100 rpm	5	5	8	19	6	6	5	5	9	21	5	10
6 rpm	1	1	2	10	2	1	1	1	3	9	2	3
3 rpm	1	1	1	7	1	1	1	1	3	6	1	2
Plastic Viscosity, cP	11	11	12	12	12	14	12	11	12	14	11	13
Yield Pt, lb/100ft^2	2	3	5	18	3	1	2	2	6	19	2	6
Gels, 10 sec/10 min.	1/1	1/1	2/2	7/8	1/2	1/1	1/2	1/2	3/4	6/6	1/2	3/3
Settling in thermocup	Y	Y	Y	N	Y	Y	Y	Y	Y	N	Y	N
HR 64 hrs at 150°F												
800 rpm at 120°F	24	28	31	54	28	28	25	25	32	58	25	35
300 rpm	12	13	18	40	15	15	13	13	20	42	13	22
200 rpm	8	9	14	34	11	11	9	9	16	36	9	13
100 rpm	5	5	9	28	7	6	5	5	11	28	5	12
6 rpm	1	1	3	19	2	1	1	1	4	15	1	4
3 rpm	1	1	2	11	2	1	1	1	3	8	1	4
Plastic Viscosity, cP	12	13	13	14	13	13	12	12	12	14	12	13
Yield Pt, lb/100ft^2	0	0	5	26	2	2	1	1	8	28	1	9
Gels, 10 sec/10 min.	1/1	1/1	2/2	14/8	2/2	1/1	1/1	1/1	4/5	7/5	2/2	5/3
Settling in Thermocup	Y	Y	N	N	Y	Y	Y	Y	Y	N	Y	N
Settling in Jar	1/4"	1/8"	1/16"	-	1/16"	1/8"	1/4"	1/4"	-	1/4"	5/16"	-
API fil, ml total	3.2	1.8	2.0	2.0	1.4	2.6	3.0	3.2	2.0	2.1	2.1	2.2
API fil, ml oil	1.6	1.6	2.0	2.0	0.4	2.6	0.8	1.2	1.8	1.8	1.4	1.9
API fil, ml brine	1.6	0.2	0.0	0.0	1.0	0.0	2.2	2.0	0.2	0.2	0.7	0.3
HTHP at 150°F, ml	8.6	6.6	8.6	8.0	9.8	13.4	11.8	14.6	7.0	4.4	12.0	5.6
HTHP at 250°F, ml			17.2		29.0				15.0	12.4	20.8	14.8
Static Age - 64 hrs at 75°F												
Total fluid height	32	32	32	32	32	32	32	32	32	32	32	32
free oil height	4	3	7	7	2	3	3	8	9	10	9	9
emulsion height	28	29	25	25	30	29	29	24	23	22	23	23
free brine height	0	0	0	0	0	0	0	0	0	0	0	0
free oil, %	13%	9%	22%	22%	6%	9%	9%	25%	28%	31%	28%	28%
emulsion, %	87.5%	90.6%	78.1%	78.1%	93.8%	90.6%	90.6%	75.0%	71.9%	68.8%	71.9%	71.9%
free brine, %	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Total	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%
MP = Multiple Phase Emulsion	MP	-	-	-	-	-						
Static Age - 24 hrs at 150°F												
Total fluid height	32	32	32	32	32	32	32	32	32	32	32	32
free oil height	7	7	10	8	10	9	4	10	10	10	10	10
emulsion height	25	25	22	24	22	23	28	22	22	22	22	22
free brine height	0	0	0	0	0	0	0	0	0	0	0	0
free oil, %	22%	22%	31%	25%	31%	28%	13%	31%	31%	31%	31%	31%
emulsion, %	78.1%	78.1%	68.8%	75.0%	68.8%	71.9%	87.5%	68.6%	68.6%	68.6%	68.6%	68.6%
free brine, %	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Total	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%
MP = Multiple Phase Emulsion	MP	-	-	-	-	-						
Difference (64 hrs/24 hrs at 150°F)												

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Cesium formate, Escaid 110 (50/5 175 ml Escaid 110 (0.803 sg) - (140.5 grams)
 175 ml Cesium Formate (2.2 sg) - (385 grams)

Mixing Procedure:

1. Measure out Escaid 110
2. Add emulsifiers, mix 5 minutes, add Barat
3. Add CsF, mix 10 min. on HB, mix on Silve
- 3A. Add Lo-Wate, mix 10 min. on HB
4. Run initial ES, viscosities at 120°F
5. Put 10 ml sample in a vial, SA for 24 hrs at 75°F, measure volumes
6. Hot-roll for 16 hours at 150°F
7. Remix, run rheology, ES, API, HTHP at 150°F
8. Put vials in oven and static-age at 150°F for 24 hrs, measure volumes
9. If 150°F HTHP is good, run at 250°F

Sample #	<u>261</u>	<u>262</u>	<u>263</u>	<u>264</u>	<u>265</u>	<u>266</u>	<u>267</u>	<u>268</u>
Integrity Sy	10	15	20	15	7	10.5	14	10.5
Rhodia Miranol CS, lb/bbl								
Witco AX-180-2, lb/bbl								
Witco DTA 350, lb/bbl dimer trimer				3				3
Baroid Bar:	50	50	50	50	50	50	50	50
Initial ES, v	285	306	355	327	8	1	1	37
Hot-rolled	320	350	314	376	2	1	0	18
Initial Properties								
600 rpm at	26	25	25	27	64	66	66	67
300 rpm	13	12	13	13	40	41	42	47
200 rpm	9	8	9	8	32	32	34	50
100 rpm	5	4	4	4	22	23	24	31
6 rpm	1	1	1	0	7	8	8	2
3 rpm	1	1	1	0	4	5	4	2
Plastic Visc	13	13	12	14	24	25	24	20
Yield Pt, lb.	0	-1	1	-1	16	16	18	27
Gels, 10 sec	0/1	0/1	1/1	1/1	6/4	6/5	6/4	5/12
Settling in t	yes	yes	yes	yes	no	no	no	no
API filt, ml	4.8	4.1	2.7	2.7	11 (1 min)	10 (1 min)	10 (1 min)	1.9
API filt, ml	-	-	-	-	-	-	-	0.6
API filt, ml	-	-	-	-	4.2	7.0	2.5	0.3
HR 16 hrs at 150°F								
600 rpm at	26	26	27	42	63	54	62	67
300 rpm	14	13	14	25	40	34	39	42
200 rpm	9	8	9	20	31	28	32	37
100 rpm	5	5	5	14	19	20	22	26
6 rpm	1	1	1	3	6	6	7	2
3 rpm	1	1	1	3	4	3	4	2
Plastic Visc	12	13	13	17	23	20	23	25
Yield Pt, lb.	2	0	1	8	17	14	16	17
Gels, 10 sec	1/1	1/1	1/1	1/1	4/5	5/4	6/5	2/9
Comments	settling	settling	settling	1/4 inch oil	3 phases	3 phases	3 phases	2 phases
24 hrs at 75°F								
Total fluid 1	32	33	33	32	32	32	32	32

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free oil height	2	2	2	2	0	0	0	12
emulsion height	28	29	29	30	21	21	23	20
free brine height	0	0	0	0	11	11	9	0
free oil, %	6%	6%	6%	6%	0%	0%	0%	38%
emulsion, %	87.5%	87.9%	87.9%	93.8%	65.6%	65.6%	71.9%	62.5%
free brine,	0.0%	0.0%	0.0%	0.0%	34.4%	34.4%	28.1%	0.0%

24 hrs at 150°F

Total fluid height	32	33	33	32	32	32	32	32
free oil height	2	4	3	5	0	0	0	12
emulsion height	30	29	30	27	21	20	22	20
free brine height	0	0	0	0	11	12	10	0
free oil, %	6%	12%	9%	16%	0%	0%	0%	38%
emulsion, %	93.8%	87.9%	90.9%	84.4%	65.6%	62.5%	68.8%	62.5%
free brine,	0.0%	0.0%	0.0%	0.0%	34.4%	37.5%	31.3%	0.0%

Difference (72 hrs/24 hrs at 150°F)

free oil, %	0%	6%	3%	9%	0%	0%	0%	0%
emulsion, %	6%	0%	3%	-9%	0%	-3%	-3%	0%
free brine,	0%	0%	0%	0%	0%	3%	3%	0%

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<u>269</u>	<u>270</u>	<u>271</u>	<u>272</u>
7	10.5	14	10.5 3
50	50	50	50
151	318	349	345
175	351	419	321
30	24	25	94
17	13	13	70
13	9	9	59
8	6	6	46
2	1	1	15
2	1	1	6
13	11	12	24
4	2	1	46
2/3	1/1	1/1	9/7
no	yes	yes	no
2.6	1/1	0.9	0.4
-	-	-	-
-	-	-	-
33	28	28	79
19	16	16	61
15	12	11	53
10	8	7	42
3	2	2	16
2	2	2	7
14	12	12	18
5	4	4	43
3/4	3/3	2/2	9/6
sagging	sagging	sagging	3/8 inch oil
32	32	32	32

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4	4	4	5
28	28	28	27
0	0	0	0
13%	13%	13%	16%
87.5%	87.5%	87.5%	84.4%
0.0%	0.0%	0.0%	0.0%

32	32	32	32
7	5	5	12
25	27	27	20
0	0	0	0
22%	16%	16%	38%
78.1%	84.4%	84.4%	62.5%
0.0%	0.0%	0.0%	0.0%

9%	3%	3%	22%
-9%	-3%	-3%	-22%
0%	0%	0%	0%

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Other embodiments of the present invention will be apparent to those skilled in the art from consideration of the present specification and practice of the present invention disclosed herein. It is intended that the present specification and examples be considered as exemplary only with a true scope and spirit of the invention being indicated by the following claims and

5 equivalents thereof.